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# Development of building material utilizing a low pozzolanic activity mineral

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## HIGHLIGHTS

- The potential use of a low pozzolanic activity mineral in producing building materials was reported.
- The mechanical properties and hydration progress of alkali activated Pisha mortar were studied.
- A maximum compressive strength of 14.4 MPa of mortar was achieved with water glass.
- The main reaction products are amorphous aluminosilicate gel (C–A–S–H) and $CaCO_3$.

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## ABSTRACT

In this work, the effects of different activators, particle size and curing conditions on the mechanical properties and hydration progress of alkali activated Pisha mortars were studied. Four different activators (NaOH, $Na_2CO_3$, $Na_2SO_4$ and water glass) based on two different fineness Pisha were used, two kinds of curing temperature, 25 and 80 °C were considerate. X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TG) and scanning electron microscopy (SEM) were conducted to analyze the reaction products, indentify the phase composition and observe the micro-morphology, respectively. It was found that the modulus of water glass and fineness of Pisha have significant influence on the compressive strength and hydration process of alkali activated Pisha mortars. The optimum activators were water glass, the sample ($M_s$ = 1.5, curing at 80 °C) exhibited the highest compressive strength (14.4 MPa) at 28 days. The results of the investigation also show that the amorphous aluminosilicate gels were the main hydration products.



## 1. Introduction

Compared to Portland cement, alkali activated materials could offer the same mechanical properties, but with lower $CO_2$ product [1–3]. The mechanical properties and economic benefit of the utilization of alkali activated materials in engineering have been well studied [4–6]. The type of activator played an important role for alkali activated material, the optimum dosage differs according to the type of aluminasilicate used and the type of activation solution [7,8].

Pisha (PS) was a special kind of pozzolanic mineral with a low activity [9], it was mainly composed of clay minerals (montmorillonite, illite and mica et.al), sand (quartz) and other minerals (feldspar, calcium, et al.) [10]. PS has a bad bonded mechanism and an unsatisfactory petrographic structure, its cementitious material, free oxide (e.g., iron oxide), is easy to dissolve in water, moreover, the structure also would be destroyed due to the expansion of soggy montmorillonite, therefore, PS is hard when it is dry, but would collapse when is immersed in water. Due to the fractured landscape and the deteriorated ecological environment, soil erosion occurs frequently during and after rainstorm, there are 20,000 tons PS per square kilometer were carried into the local river every year [11], and the PS was considerate as one of the main sources of sediment into the local river. However, there is rarely researchers pay their attention to study the exploitation of PS. In fact, PS could be reused to produce alternative binding materials for dam building and other civil engineering [9]. Therefore, it would be a beneficial exploration to apply PS in the construction industry (e.g., dam building materials or building brick) as a pozzolanic material for the production of alkali activated materials.

The aim of this paper is to produce a new alkali activated building materials by using PS, and study the effect of alkaline type and

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the fineness of PS at different curing conditions on the geopolymerisation of PS. It is hoped that the processing of alkali activated Pisha martors (APSM) could be well understood and found the optimal scheme of manufacture APSM by using PS.

## 2. Materials and methods

### 2.1. Materials

The PS was obtained from Loess Plateau in Inner Mongolia, China. The PS was air-dried and pulverized to give a granular material with particles less than 1.0 mm in diameter, and part of the PS (clay mineral, sand and all the other phases) was dry milled using a porcelain ball mill with alumina milling media for 30 min to increase the specific surface area. The particle size distributions of PS and milled PS shown in Fig. 1 were determined using laser diffraction (Mastersizer 2000, Malvern Instruments). For PS and milled PS, Fig. 1 indicates a mean particle size of approximately 111 and 18.9 μm, respectively. The $d_{90}$ (90% of volume less than this size) of PS and milled PS was found to be 309.6 and 52.6 μm, and the value of $d_{10}$ was fund to be 8.52 and 2.68 μm, respectively.

The chemical composition of PS showed in Table 1 was determined by X-ray fluorescence (XRF) spectrometry (Germen, Siemens-Bruker, SRS 3400). The activator solution was prepared by dissolving NaOH, $NaCO_3$ and $Na_2SO_4$ pellets (99% purity quotient, Tianjin Kemiou Chemical Reagent Co., Ltd. China) in distilled water to a certain concentration, and cooled to room temperature. Water glass with $M_s$ (molar ratio $SiO_2/Na_2O$) = 3.0 ($Na_2O$ = 8.83 wt%, $SiO_2$ = 26.5 wt%, $H_2O$ = 64.67 wt%) was used as alkaline activator, and the others were a mixture of this commercial sodium silicate with NaOH solution to give a combined modulus of $M_s$ = 2.0 ($Na_2O$ = 13.25 wt% $SiO_2$ = 26.5 wt%, $H_2O$ = 60.25 wt%) and $M_s$ = 1.5 ($Na_2O$ = 17.67 wt% $SiO_2$ = 26.5 wt%, $H_2O$ = 55.83 wt%), respectively. The pH value of the activator solution was measured using a pH meter (METER TOLEDO, Delta 320, LAB, Switzerland) after the activator solution was prepared.

### 2.2. Sample preparation

The materials used to prepare the mortars were summarized in Table 2. For all samples, the total mass of the mixture was kept at 392.5 g. Add the PS powder into activator solution (e.g., NaOH, $NaCO_3$, $Na_2SO_4$, water glass), in order to achieve complete mixing between the solid and solution, the mixture was mixed for 15 min by a magnetic stir bar. To make regularly shaped specimens for mechanical testing, the mixtures were poured into cylindrical steel molds, and mixture was pressed (30 kN) to specimens with an diameter of 5 cm and height of 10 cm (i.e., an aspect ratio of 2.0) bar on a hydraulic testing machine. To ensure repeatability, 3 specimens were prepared for each type of mortar. To investigate the effect of curing temperature on strength of APSM, the specimens were sealed with plastic bags and cured in oven (80 °C) and laboratory at ambient temperature (25 ± 2 °C), respectively.

### 2.3. Methods

The compressive strength was tested by using an electronic universal testing machine with a 100 kN capacity and a constant displacement rate of 0.05 mm/min. The composition of raw materials and mortars were tested by X-ray diffraction (XRD), XRD was recorded on a Siemens-Bruker D5000 using Cu Kα radiation (λ = 1.54 Å) operating at 40 kV and 30 mA. The samples were scanned from 5 to 70° (2θ range) at a rate of 2°/min and step size of 0.02°. Fourier transform infrared (FTIR) analysis was performed using the KBr pellet method (1 mg sample per 100 mg KBr) on a Bruker EQUINOX 55 spectrometer, with 32 scans per sample collected from 4000 to 400 $cm^{-1}$ at 4 $cm^{-1}$ resolution. A Switzerland Mettler-Toledo simultaneous thermal analyser was used to measure some physical properties of the material as a function of the temperature change. The samples were heated

**Table 1**
Chemical composition of PS determined by X-ray fluorescence.

| Chemical | Component (wt%) |
|---|---|
| $SiO_2$ | 62.46 |
| $Al_2O_3$ | 20.08 |
| CaO | 5.10 |
| $Na_2O$ | 0.56 |
| $K_2O$ | 2.23 |
| MgO | 5.02 |
| $Fe_2O_3$ | 3.10 |
| $SO_3$ | 0.04 |
| LOI | 1.41 |

LOI is loss on ignition at 1000 °C. Att values in wt%.

**Table 2**
Mix proportion of mixture (wt%).

| Sample | PS | Activator solution | | | | Curing temperature °C | PS type |
|---|---|---|---|---|---|---|---|
| | | Activators | | Water | pH | | |
| W1-1 | 84 | Water glass ($M_s$ = 1.5) | 3 | 13 | 11.5 | 80 | PS |
| W1-2 | | | | | | 25 | PS |
| W1-3 | | | | | | 80 | Milled PS |
| W1-4 | | | | | | 25 | Milled PS |
| W2-1 | 84 | Water glass ($M_s$ = 2.0) | 3 | 13 | 10.7 | 80 | PS |
| W2-2 | | | | | | 25 | PS |
| W2-3 | | | | | | 80 | Milled PS |
| W2-4 | | | | | | 25 | Milled PS |
| W3-1 | 84 | Water glass ($M_s$ = 3.0) | 3 | 13 | 9.0 | 80 | PS |
| W3-2 | | | | | | 25 | PS |
| W3-3 | | | | | | 80 | Milled PS |
| W3-4 | | | | | | 25 | Milled PS |
| NC1 | 86 | $Na_2CO_3$ | 1 | 13 | 8.8 | 80 | PS |
| NC2 | | | | | | 25 | PS |
| NC3 | | | | | | 80 | Milled PS |
| NC4 | | | | | | 25 | Milled PS |
| NS1 | 86 | $Na_2SO_4$ | 1 | 13 | 7.4 | 80 | PS |
| NS2 | | | | | | 25 | PS |
| NS3 | | | | | | 80 | Milled PS |
| NS4 | | | | | | 25 | Milled PS |
| NH1 | 86 | NaOH | 1 | 13 | 10.9 | 80 | PS |
| NH2 | | | | | | 25 | PS |
| NH3 | | | | | | 80 | Milled PS |
| NH4 | | | | | | 25 | Milled PS |

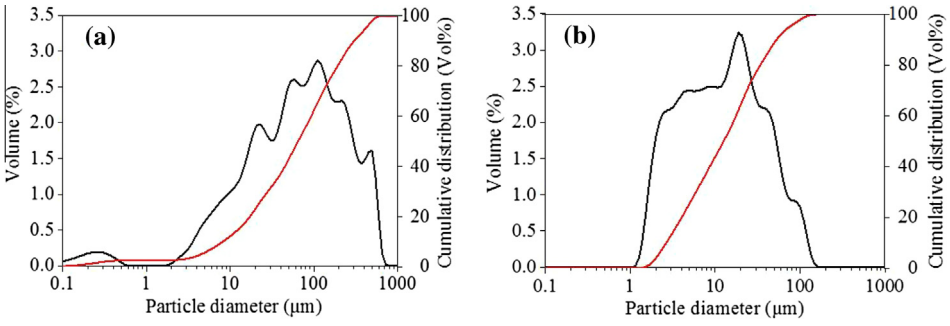


**Fig. 1.** Particle size distribution curve of raw materials. (a) PS, (b) milled PS.